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Mechanical Testing Data from Neutron Irradiations of PM-HIP and Conventionally Manufactured Nuclear Structural Alloys

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Data Article

Mechanical testing data from neutron irradiations of PM-HIP and conventionally manufactured nuclear structural alloys

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a r t i c l e i n f o

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a b s t r a c t

This article presents the comprehensive mechanical testing data archive from a neutron irradiation campaign of nuclear structural alloys fabricated by powder metallurgy with hot isostatic pressing (PM-HIP). The irradiation campaign was designed to facilitate a direct comparison of PM-HIP to conventional casting or forging. Five common nuclear structural alloys were included in the campaign: 316L stainless steel, SA508 pressure vessel steel, Grade 91 ferritic steel, and Nibase alloys 625 and 690. Irradiations were carried out in the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL) to target doses of 1 and 3 displacements per atom (dpa) at target temperatures of 300 and 400 \degree C. This article contains the data collected from post-irradiation uniaxial tensile tests following ASTM E8 specifications, fractography of these tensile bars, and nanoindentation. By making this systematic and valuable neutron irradiated mechanical behavior dataset openly available to the nuclear materials research community, researchers may now use this data to populate material performance databases, validate material

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performance and hardening models, design follow-on experiments, and enable future nuclear code-qualification of PM-HIP techniques.

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Specifications Table

Value of the Data

- Data represent a comprehensive mechanical characterization of unique neutron irradiated powder metallurgy - hot isostatically pressed (PM-HIP) structural alloys, with a direct comparison to the cast or forged counterpart alloy. Direct comparisons of advanced manufactured materials to their conventionally manufactured counterparts – particularly under neutron irradiation – are rare.
- Neutron irradiation experiments are costly, time-consuming, and challenging, so the resultant data can offer considerable value to the nuclear materials and irradiation effects research communities.
- Historical data from neutron irradiated materials (especially legacy power plant materials) have been poorly archived in the open, accessible literature. By making the data herein open and permanently accessible, we aim to contribute to growing this critical database.
- Researchers working in irradiation effects and nuclear reactor materials may be able to use these data to enhance our overall understanding of mechanical behavior of structural alloys under neutron irradiation, and guide future mechanistic experiments.
- Data can support future nuclear code qualification efforts for PM-HIP alloys.
- Data can be used for verification and validation of predictive mechanics models for nuclear structural material lifetimes and performance, including use as training data for machine learning models.

1. Objective

The objective is to publish the mechanical behavior data from neutron irradiated structural alloys fabricated by both powder metallurgy with hot isostatic pressing (PM-HIP) and conventional casting or forging. Neutron irradiations with post-irradiation examination (PIE) are required to qualify advanced materials for nuclear reactor service. But these neutron irradiation and PIE campaigns are time-consuming, often spanning 7 or more years, and can cost multiple millions of dollars partially because of the precautions necessary for characterizing radioactive specimens. Hence, data generated from these campaigns is of tremendous value to the nuclear materials and irradiation effects research communities. Here, we publish a comprehensive neutron irradiated mechanical testing dataset that enables direct comparisons of PM-HIP alloys to their cast/forged counterparts. Alloys studied are 316L stainless steel, SA508 pressure vessel steel, Grade 91 ferritic steel, and Ni-base alloys 625 and 690. Irradiations were carried out in the Advanced Test Reactor (ATR) to target doses of 1 and 3 displacements per atom (dpa) at target temperatures of 300 and 400 °C. Mechanical data included are ASTM E8 uniaxial tensile tests, fractography, and nanoindentation. This data can be used to populate material performance databases, validate models, design follow-on experiments, and enable future code-qualification of PM-HIP manufacturing.

2. Data Description

Nanoindentation, uniaxial tensile testing, and fractography are conducted on five nuclear structural alloys fabricated by PM-HIP and a conventional method (specifically, casting or forging), then irradiated to a range of doses and temperatures. The specific forms of data are:

Tensile Data – Raw data are provided in .csv format. Each data file corresponds to a single tensile specimen. Files contain a header with metadata including the specimen name, testing conditions, and experiment date and operator. The remainder of each file contains the tabulated measured stress-strain curve.

Fractography Data – Files are organized into folders by specimen (i.e., broken tensile bar). Multiple image files are collected from each specimen. Typically, an "overview" image is provided, with numerous additional higher-magnification images of representative areas on the fracture surface. All fractographs are provided in software-agnostic .jpg, .bmp, or .tiff format.

Nanoindentation Data – Files are organized into folders by specimen. Each folder contains three file types:

- (1) One "Read Me" .txt file: The first line of these files lists the total number of indents, N, made on the given specimen (typically $N = 20-30$ indents per specimen). The remainder of this file lists the file names corresponding to each of these N indents and tabulates key indentation results and parameters, specifically: maximum indent depth h_{max} (nm), characteristic depth h_c (nm), maximum load P_{max} (μ N), stiffness S (μ N/nm), indent area A $(nm²)$, effective depth h_{eff} (nm), reduced modulus E_r (GPa), and hardness H (GPa). These values can be used with the corresponding raw data files (see #2 below) to calculate H versus depth and E versus depth curves, per the Oliver-Pharr method.
- (2) A total of N raw data .txt files, each corresponding to one of the indents made on the given specimen. Indent depth (nm), load (μN) , and time (s) are tabulated throughout the duration of each indent, from initial to deepest indent depths. Load-displacement curves can be generated from these raw data.
- (3) Some specimen ID numbers will have an associated .jpg image that displays the array of indents made on the specimen. Images were not collected for all specimens, so are not available for all specimens (i.e., in all folders).

3. Experimental Design, Materials and Methods

This article presents a complete dataset from the mechanical testing conducted as part of a neutron irradiation campaign $[1-3]$ aiming to directly compare the irradiation behavior of five PM-HIP [\[4–6\]](#page-9-0) nuclear structural alloys to their conventional cast or forged counterparts. The intention of this article is to make this high-value, systematic neutron irradiated mechanical behavior dataset openly and permanently available to the nuclear materials community for research and practical purposes such as populating material performance databases, validating hardening models, designing follow-on experiments, and providing materials qualification data.

3.1. Materials and Irradiations

Five common nuclear structural alloys were studied, specifically 316L stainless steel, SA508 Grade 3 Class 1 pressure vessel steel, Grade 91 ferritic steel, and two Ni-based alloys 625 and 690. All five alloys had a PM-HIP version and a conventionally manufactured (cast or forged) version. Alloy compositions measured by ICP-AES and processing parameters are provided in [Tables](#page-5-0) 1 and [2,](#page-5-0) respectively. The unirradiated thermal and mechanical behaviors of Alloys 625, 690, and 316L have been documented in Refs. [\[7–9\]](#page-9-0) and the ion irradiation response of Alloy 625 in Ref. [\[10\].](#page-10-0) From each alloy, specimens were machined into either transmission electron microscopy (TEM) discs or round tensile bars, [Fig.](#page-5-0) 1. The TEM discs were cut by wire electrical

Table 1 Alloy compositions in wt%, measured by ICP-AES.

Allov	Fabrication C		Si	Mn	\mathbf{P}	S	Сr	Ni	Mo	Ti	C ₁₁	Al	Co	Ph	Fe	v	Nb N	
625	PM-HIP	0.01				0.45 0.41 0.003 0.003 21.9 Bal			8.2					0.006 < 0.1 < 0.05 < 0.1 < 0.010 3.6				
625	Forged	0.01				0.20 0.42 0.006 0.004 23.7 Bal			7.6	0.31	$\overline{}$	0.02	$\overline{}$		3.5°	$\overline{}$	$3.6 -$	
690	PM-HIP	0.019	0.45 0.37 -			0.003 30.9 Bal			$\overline{}$	\equiv	0.01	<0.02 –			9.6	$\overline{}$		
690	Forged	$\overline{}$		$0.12 \quad 0.59 -$		0.003	31.3 Bal		$\overline{}$	0.31	$\overline{}$	0.26	$\overline{}$		$10.3 -$			
Grade 91	PM-HIP	0.12				0.47 0.62 0.004 0.003 8.78 0.08 0.92 -					\equiv	0.12	$\overline{}$	$\overline{}$	Bal	0.27	$\overline{}$	
Grade 91	Cast	0.10				0.24 0.48 0.004 0.008 8.40 0.09 0.86 -					\equiv	0.05	$\overline{}$	$\overline{}$	Bal	$0.20 -$		
SA 508	PM-HIP	0.01				0.21 1.39 0.002 0.005 0.18 0.79 0.37 -									Bal	$\overline{}$		
SA 508	Forged	0.02				0.31 0.46 0.003 0.007 0.21 0.50 0.26 -									Bal	0.01	$\overline{}$	
316L	PM-HIP	0.004 0.88 1.41			0.005 0.005		17.6	$125210 -$							Bal	\equiv	\equiv	< 0.1
316L	Forged	0.03		0.52 1.52		$0.045 \le 0.03$ 16.3		10.7	$1.86 -$		0.35	$\overline{}$			Bal	\equiv	-	< 0.1

Table 2

Alloy processing parameters [\[18\].](#page-10-0)

(b) Tensile Bars

Fig. 1. Specimen geometries of (a) TEM discs and (b) tensile bars (all dimensions in inches).

specimens.

Table 4

List of files for 316L stainless steel specimens.

Fabric ation	Specimen ID Type	Specimen	Target Dose [dpa]	[°C]	Target Temp Actual Dose [dpa]	Actual Avg Temp $[°C]$	Nanoi ndent	Tensile	Fracto- graphy
Cast	643 704 705	TEM Disc Tensile Tensile	3	400 400 400	3.83 3.84 3.94	396 379 382	Yes $\overline{}$	Yes Yes	$\overline{}$
PM-HIP	646 719 720	TEM Disc Tensile Tensile		400 400 400	4.00 3.91 3.77	397 380 373	Yes	Yes Yes	Yes

discharge machining (EDM) to 3 mm in diameter and ∼250 μm thick. They were subsequently hand polished using successively finer diamond suspensions (to the finest suspension of 1 μm diamond) to achieve a mirror finish and a final disc thickness of 150 μm for compliance with dimensions shown in [Fig.](#page-5-0) 1. The tensile specimen geometries were guided by ASTM standard E8, having a total length of 76.2 mm (3 in) and gauge diameter of 6.35 mm (0.25 in). The tensile specimens were cut by computer numerical control (CNC) machining to a surface roughness of 3.2 μm. Each specimen was engraved with a unique identification (ID) number.

Following machining and dimensional inspections, specimens were loaded for irradiation experiments into non-instrumented stainless steel capsules, which acted as sealed pressure boundaries between the specimens and the reactor coolant. Capsules were pressurized with He or Ar gas to control temperature; quartz-encapsulated melt wires in capsules were used to estimate in-pile temperature history. Capsules were then assembled into test trains for reactor insertion. Additionally, the irradiation experiment design process included structural, thermal, and neutronic analyses as described in Refs. [\[11,12\]](#page-10-0). A comprehensive description of the capsule design, assembly, and irradiation experiment design is provided in Ref. [\[1\].](#page-9-0)

The test trains were loaded into Advanced Test Reactor (ATR) inboard positions A-6, A-7, and A-8. The irradiation occurred during ATR cycles 164A, 164 B, 166A and 166B, between May 9, 2018, and January 10, 2020. The estimated total irradiation dose was calculated using MCNP5 release 1.60 [\[13,14\]](#page-10-0) using microscopic cross-section data generated by NJOY [\[15\].](#page-10-0) Specimen maximum and average temperatures during irradiation were estimated by finite-element analysis in ABAQUS, together with the melt wire analysis, as described in refs. [\[16,17\]](#page-10-0). As-run thermal and dose analysis is comprehensively described in Ref. [\[1\].](#page-9-0)

The actual neutron irradiation doses ranged \sim 0.5–4.5 displacements per atom (dpa) and actual specimen average temperatures ranged ∼260–400 °C. The specific dose and temperature history of each specimen is listed in Tables 3–8, which are organized by alloy. The type of mechanical testing data obtained from each specimen – i.e., nanoindentation, uniaxial tensile testing, and/or fractography – is also specified in Tables 3–8.

3.2. Mechanical Testing of Irradiated Specimens

After neutron irradiation, all specimens are unloaded from their capsules, decontaminated, and sorted within the radioactive material handling host cells at the Hot Fuel Examination Facility (HFEF) at Idaho National Laboratory (INL). From there, tensile specimens were moved within

Table 6

List of files for Alloy 690 specimens.

Fabric ation	Specimen ID	Specimen Type	Target Dose [dpa]	Target Temp [°C]	Actual Dose [dpa]	Actual Avg Temp \lceil ^o C \rceil	Nanoi ndent	Tensile	Fracto- graphy
Forged	Unirrad	Tensile	Ω	Ω	Ω	Ω		Yes	
	415	TEM Disc	1	400	1.09	389	Yes		
	518	Tensile		400	0.99	358		Yes	
	519	Tensile		400	0.86	335	-	Yes	
	301	Tensile	3	300	3.79	284	-	Yes	
	302	Tensile	3	300	4.13	306		Yes	
	627	TEM Disc	3	400	4.11	398	Yes		
	601	Tensile	3	400	3.12	373		Yes	
	602	Tensile	3	400	3.52	385	-	Yes	
PM-HIP	Unirrad	Tensile	Ω	Ω	Ω	Ω		Yes	
	408	TEM Disc		400	1.09	388	Yes		
	457	Tensile		400	0.99	342	-	Yes	
	458	Tensile		400	0.86	368		Yes	
	344	Tensile	3	300	3.96	288		Yes	
	345	Tensile	3	300	3.53	266		Yes	
	621	TEM Disc	3	400	4.14	398	Yes	-	
	673	Tensile	3	400	3.29	378	-	Yes	
	674	Tensile	3	400	2.82	378		Yes	

the HFEF hot cells for uniaxial tensile testing, while the TEM disc specimens were packaged for shipping to the Center for Advanced Energy Studies (CAES) for nanoindentation testing.

Uniaxial tensile data was collected using a 13M Instron load frame located inside the INL HFEF hot cells. Although this load frame is dedicated for radioactive materials, the unirradiated tensile bars were also tested on this same load frame for consistency and to eliminate effects of instrument variability. Testing was conducted in accordance with ASTM standard E8 for threaded grip specimens. All tensile testing was conducted at ambient temperature in an Ar environment. Strain rate was 8.78 \times 10⁻³ s⁻¹, which corresponds to a crosshead speed of 0.279 mm/min. After 10% strain, the strain rate was increased to 0.0315 s⁻¹ (crosshead speed 1.0 mm/min) until failure.

Following tensile testing, some specimens of interest were selected for fractography. To prepare specimens for fractography, ∼1-2 mm (including the fracture surface) was cut off the end

Fabri cation	Specimen ID	Specimen Type	Target Dose [dpa]	[°C]	Target Temp Actual Dose [dpa]	Actual Avg Temp \lceil ^o C \rceil	Nanoi ndent	Tensile	Fracto- graphy
Cast	Unirrad	Tensile	0	0	Ω	Ω		Yes	
	425	TEM Disc	1	400	0.99	389	Yes	-	
	503	Tensile		400	1.00	386	-	Yes	Yes
	504	Tensile		400	0.99	376	-	Yes	
	666	TEM Disc	3	400	3.97	397	Yes	-	
	605	Tensile	3	400	3.71	363	$\qquad \qquad -$	Yes	
	703	Tensile	3	400	3.54	362	-	Yes	Yes
PM-HIP	Unirrad	Tensile	0	0	Ω	Ω		Yes	
	424	TEM Disc	1	400	0.99	389	Yes		
	403	Tensile		400	1.02	367	$\qquad \qquad -$	Yes	Yes
	404	Tensile		400	1.01	358		Yes	
	636	TEM Disc	3	400	3.74	397	Yes		
	605	Tensile	3	400	3.71	363	-	Yes	
	670	Tensile	3	400	3.68	362	-	Yes	Yes
	721	Tensile	3	400	3.40	351		Yes	

Table 8

List of files for SA508 steel specimens.

Fabri cation	Specimen ID	Specimen Type	Target Dose [dpa]	\lceil °C]	Target Temp Actual Dose [dpa]	Actual Avg Temp $[°C]$	Nanoi ndent	Tensile	Fracto- graphy
Forged	Unirrad	TEM Disc	Ω	0	Ω	Ω	Yes		
	Unirrad	Tensile	$\bf{0}$	0	Ω	Ω	-	Yes	
	110	TEM Disc	$\mathbf{1}$	300	0.69	286	Yes		
	114	TEM Disc	$\mathbf{1}$	300	0.69	286	Yes	-	
	201	Tensile	1	300	0.53	265		Yes	
	206	Tensile		300	0.83	270		Yes	
	437	TEM Disc	1	400	0.95	384	Yes	-	
	501	Tensile		400	0.96	362	$\overline{}$	Yes	
	502	Tensile	1	400	0.98	385	-	Yes	Yes
PM-HIP	Unirrad	TEM Disc	$\bf{0}$	0	Ω	Ω	Yes		
	Unirrad	Tensile	0	0	0	0	-	Yes	
	104	TEM Disc	1	300	0.70	286	Yes		
	101	Tensile		300	0.54	266	-	Yes	
	127	Tensile	1	300	0.84	273	-	Yes	
	434	TEM Disc	$\mathbf{1}$	400	0.98	388	Yes	-	
	401	Tensile		400	0.97	343	-	Yes	Yes
	402	Tensile		400	1.00	365		Yes	

of one of the broken "halves" of the tensile bar. Cutting was done with a low-speed saw, and the cut was made normal to the tensile axis. This allowed for the fracture surface to be accommodated face-up within a scanning electron microscope (SEM) for fractography, and also minimized the radioactive material volume within the SEM. The cutting was done in the hot cells in HFEF, then specimens were subsequently moved into the Lyra3 Tescan SEM, also at HFEF at INL. SEM images were collected in secondary electron (SE) mode. An overview image of the entire fracture surface was taken for each specimen of interest. Additional images were taken to show representative features of the fracture surface in higher magnification and detail.

Nanoindentation was conducted on the TEM disc specimens shipped to CAES. Their surfaces were first prepared by jet electropolishing in a 90% methanol $+ 10\%$ perchloric acid solution maintained at -20 °C. A Berkovich indenter tip was used in continuous stiffness mode. For the SA508 specimens, nanoindentation was conducted in load-controlled mode to a maximum load of 8000 μN with a loading time of 5 s, holding time of 5 s, and unloading time of 5 s. For all other alloys, nanoindentation was conducted in depth-controlled mode to a maximum depth of 3500 nm at a strain rate of 0.2 s⁻¹. Indents were generally made in 6 \times 5 indent arrays. All nanoindentation was conducted using a Hysitron TI-950 TriboIndenter at CAES operating in ambient temperature and atmosphere.

Ethics Statements

This work did not involve human subjects, animal experiments, or data collected from social media platforms.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

PM-HIP [Mechanical](https://doi.org/10.17632/9z98kdkpyz.1) Data Archive (Original data) (Mendeley Data).

CRediT Author Statement

Janelle P. Wharry: Conceptualization, Project administration, Supervision, Funding acquisition, Writing – original draft; **Caleb D. Clement:** Investigation, Data curation, Writing – review & editing; **Yangyang Zhao:** Investigation, Data curation, Writing – review & editing; **Katelyn Baird:** Investigation, Writing – review & editing; **David Frazer:** Investigation, Writing – review & editing; **Jatuporn Burns:** Investigation, Writing – review & editing; **Yu Lu:** Investigation, Writing – review & editing; **Yaqiao Wu:** Investigation, Writing – review & editing; **Collin Knight:** Project administration, Supervision, Writing – review & editing; **Donna P. Guillen:** Project administration, Supervision, Funding acquisition, Writing – review & editing; **David W. Gandy:** Conceptualization, Project administration, Funding acquisition, Writing – review & editing.

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